JP S 60-186479   2-1-8

METHOD OF PRODUCING HIGH-HEAT CONDUCTIVE ALUMINUM NITRIDE SINTERED BODY

2. Claims

1. A method of producing a high-heat conductive aluminum nitride sintered body comprising:

sintering a blended powder prepared by adding carbon or a compound to decompose into carbon in an amount of 0.05% by weight or more to 2% by weight or less in terms of carbon to an AlN powder having a mean particle diameter of 3 μm or less.

2. The method of producing a high-heat conductive aluminum nitride sintered body according to claim 1, wherein the sintering is conducted in the temperature range of 1,700°C to 2,100°C and is pressure sintering at a pressure of 50 Kg/cm^2 or more.

3. Detailed Description of the Invention

[Technical Field of the Invention]

The present invention relates to a method of producing an aluminum nitride sintered body possessing high-heat conductivity.

[Technical Background of the Invention and Problems to be Solved]

Aluminum nitride is known as ceramics excellent in insulating properties and thermal conductivity.

However, the raw material aluminum nitride powder for an aluminum nitride sintered body usually contains impurities including oxygen. This causes the problem of the thermal conductivity of the sintered body obtained being at most about 40 to 45 w/m·K even though the raw material powder is sintered in a step wherein the mixing of impurities is prevented as much as possible, for example by means of pressure sintering at 1,600°C to 1,900°C, and better high-heat conductivity is required.

[Purpose of the Invention]

The object of the present invention is to solve the above-mentioned problem and provide a method of producing an aluminum nitride sintered body excellent in thermal conductivity.

[Summary of the Invention]

The present inventors tried to attain the above-mentioned object and found out that it is extremely effective to subject to pressure sintering a blended powder prepared by adding 0.05% to 2% by weight of carbon or a compound to decompose into carbon and produce the same amount of carbon to an aluminum nitride powder having a mean particle diameter of 3 μm or less.

While the higher sintering pressure is better, it is preferable that the sintering pressure is substantially in the range of 50 to 600 Kg/cm^2 in view of densification of the sintered body, improvement of the thermal conductivity and the strength of a vessel for use in sintering.

Rendering the sintering temperature to be below 1,700°C does not bring about densification of a sintered body; on the other hand, making the temperature exceed 2,100°C does not provide a good sintered body due to increased evaporation of aluminum nitride, and thus the temperature is set in the range of 1,700°C to 2,100°C. An excessive amount of carbon to be added causes carbon to remain in the sintered body, which becomes a cause of dropping the thermal conductivity, and therefore the amount needs to be in the range of 0.05 to 2% by weight. In addition, rendering the size of AlN powder to exceed 3 μm lowers denseness and thermal conductivity, and so it is regulated to be 3 μm or less.

[Advantages of the Invention]

Regarding an aluminum nitride sintered body of the present invention, the thermal conductivity of the sintered body is able to be improved by a simple operation of sintering a blended powder prepared by adding a predetermined amount of carbon or compound to decompose into carbon to an aluminum nitride powder. In addition, because carbon is the only additive and the carbon added removes the oxygen contained in the aluminum nitride powder to allow the sintered body after sintering to be very highly pure, it can be expected the possibility of application to an aluminum melting crucible, which needs to be highly pure.

[Examples of the Invention]

The invention will be described in detail by means of

examples below.

Example 1

To 100 g of an aluminum nitride powder having a mean particle diameter of 3 μm or less was added 0.1 g of amorphous carbon (particle diameter 1 μm or less) and the resulting material was wet mixed for 4 hours by means of a ball mill. After drying, it was subjected to pressure forming at a pressure of 300 Kg/cm^2 using a mold press to obtain a green compact of about 30 mm × 30 mm × 12 mm.

The green compact placed in a carbon mold was subjected to pressure sintering at 300 Kg/cm^2 and 1,800°C for one hour in a nitrogen gas atmosphere. Disks having a diameter of 10 mm and a thickness of 4 mm were cut out of the sintered body thus obtained. The thermal conductivity was determined to be 56 w/m·K by the laser flash method. The density of the sintered body was 3.26 g/cm^3.

Examples 2 to 9

By using an aluminum nitride powder and carbon similar to those in Example 1 as well as various carbon concentrations and sintering temperatures and pressures, aluminum nitride sintered bodies were prepared. The thermal conductivities thereof were measured and the results are shown in Table 1.

Table 1

Example No.	Carbon concentration (% by weight)	Sintering conditions			Sintered body density (g/cm^3)	Thermal conductivity (w/m·K)
		Temperature (°C)	Pressure (kg/cm^2)	Time		

2	0.05	1800	300	1	3.26	50
3	0.1	1700	400	2	3.24	52
4	0.1	1900	300	0.5	3.26	55
5	0.2	1800	200	1	3.25	49
6	0.4	1850	300	1	3.26	47
7	0.6	1800	300	1	3.24	47
8	0.8	1800	300	1	3.22	45
9	1.0	1800	300	1	3.21	45
10	0.1	2100	300	1	3.26	56

Example 11

To 100 g of aluminum nitride powder having a mean particle diameter of 2 μm or less was added 1 g of sugar as a carbon source and the resulting material was dry mixed for 24 hours by a ball mill. This blended powder was subjected to pressure forming at a pressure of 300 Kg/cm^2 using a mold press to obtain a green compact of about 30 mm × 30 mm × 12 mm. The green compact was placed in a carbon mold and was subjected to pressure sintering at 300 Kg/cm^2 and 1,800°C for 30 minutes in a nitrogen gas atmosphere. Disks having a diameter of 10 mm and a thickness of 4 mm were cut out of the sintered body thus obtained. The thermal conductivity was determined to be 55 w/mK by the laser flash method. The density of the sintered body was 3.26 g/cm^3.

Comparative Examples 1 to 5

By using an aluminum nitride powder and carbon similar to those in Examples 1 to 9 as well as various carbon concentrations and sintering temperatures and pressures, aluminum nitride sintered bodies were prepared. Thermal conductivities thereof were measured and the results are shown in Table 2.

Comparative Example No.	Carbon concentration (% by weight)	Sintering conditions Temperature (°C)	Pressure (kg/cm^2)	Time	Sintered body density (g/cm^3)	Thermal conductivity (w/m·K)
1	0	1800	300	1	3.24	41
2	4	1800	300	1	3.00	35
3	10	1800	300	1	2.75	27
4	0	1950	200	1	3.20	37